Compost Age and Sample Storage Effects on Maturity Indicators of Biosolids Compost

T. A. Butler, L. J. Sikora,* P. M. Steinhilber, and L. W. Douglass

ABSTRACT

Compost product safety and quality assurance are required to meet the needs of the horticultural, agricultural, and silvicultural user markets. At present, there exist no industry-wide sampling and testing protocols for compost products, thus limiting the production sector. The objective of this research was to test three methods for determining compost maturity. The study followed the composting process of a locally successful commercial composting operation that had been producing lime-stabilized biosolids compost in the Washington, DC metro region for 12 yr. Change over time in the dependent variables—Dewar flask self-heating capacity, oxygen uptake rate, and cation exchange capacity (CEC)—during a 57-d composting of lime-stabilized biosolids was studied. Because cold storage at 4°C is recommended when compost samples cannot be tested for maturity immediately, cold storage of up to 11 wk was included as a variable. Mathematical models were developed that predict change in the Dewar flask self-heating capacity, oxygen uptake rate, and CEC with composting time and storage at 4°C. The Dewar flask self-heating test was the most useful indicator of compost maturity. This test showed change throughout the 57-d biosolids composting period while oxygen respirometry did not change after 29 d. The CEC was found to increase with age and storage. Storage effects varied for the different tests. Except for Days 1 and 57, composts continued to stabilize during storage. Testing stored composts may produce erroneous results that suggest the compost is mature.

THE composting industry, including producers, marketers, testing laboratories, and consumers have a well-documented need to know the specific chemical, physical, and biological properties of compost products (Leege, 1996). Composting has become a preferred method for municipalities and industries to recycle a variety of organic byproducts, transforming them into useful soil conditioners and amendments. Because technologies for composting and recycling vary, a wide spectrum in the quality of the finished products exists. The development of uniform standards is necessary so that users in many markets nationwide can use compost safely for specific applications.

Aerobic composting is the biological oxidative decomposition of organic materials by successive communities of microorganisms under different temperature regimes, which produce a humified end-product. The basic principles of the composting process have been discussed extensively in the literature (Gouleke, 1972; Haug, 1980; Polprasert, 1989, p. 63–80; Miller, 1994; Stratton et al., 1995). The degree of completion of the composting process can be evaluated by measuring various changes in the chemical, physical, and biological properties of the substrate. Although for the purposes of discussion the term "compost maturity" can be used generically to refer to the degree of completion of the composting process, the technical terms "compost maturity" and "compost stability" are not synonymous (Iannotti et al., 1993). Compost maturity refers to the degree of humification of the material. Compost stability refers to the level of activity of the microbial biomass. The degree of compost maturity at the conclusion of the process is critical to the marketability of the product.

One problem associated with immature compost is continued decomposition. The continued decomposition of an immature compost in soil can induce anaerobic conditions as the microbial biomass utilizes oxygen in the soil pores to break down the material. This in turn can deprive plant roots of oxygen, and lead to the generation of hydrogen sulfide (H_2S) and nitrite (NO_2^-) (Mathur et al., 1993). Another important problem associated with the application of immature composts to soils is the prevalence of a high (25:1 or greater) carbon to nitrogen ratio in such materials (Iglesias Jimenez and Perez Garcia, 1989). Immature composts with a high C to N ratio can induce nitrogen starvation in plants as microbes scavenge soil N to make up the deficit. Other problems include phytotoxicity due to the presence of organic acids as the intermediate by-products of continuing decomposition. Acetic acid and phenolic compounds, in particular, may suppress seed germination, inhibit root growth, or suppress crop yields (Zucconi et al., 1981a,b; Chanyasak et al., 1983a,b; Hadar et al., 1985).

Cation exchange capacity (CEC) has been heavily investigated as a potential indicator of compost maturity. The CEC in an organic material increases as a function of humification due to the formation of carboxyl and phenolic functional groups (Lax et al., 1986). Carboxyl groups are formed by the oxidation of lateral chains of the aromatic rings or the hydrolysis of esters or lactones (Lax et al., 1986). Harada and Inoko (1980) developed a method for determining the CEC of municipal refuse compost as an indicator of its maturity. These researchers considered a municipal refuse compost with a CEC of greater or equal than 60 cmol kg^{-1} expressed on an ash-free basis to be sufficiently mature for application to the soil. Harada et al. (1981) found a strong negative correlation between CEC and the solid C to N ratio of municipal refuse compost ($r = -0.94$). Saharinen (1998) reported that CEC should be calculated per unit of ash since this prevents the overestimation of the number of cation exchange sites on composted materials.

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Abbreviations: BVS, biological volatile solids; CEC, cation exchange capacity; VS, volatile solids; WSSC, Washington Suburban Sanitary Commission.

Other researchers have proposed microbial respiration rate as a potential indicator. Willson and Dalmat (1986) proposed an oxygen uptake rate of 20 mg kg^{-1} h^{-1} as an acceptable criterion of maturity. Iannotti et al. (1993) found an initial high rate of oxygen consumption (2.0 mg g^{-1} volatile solids [VS] h^{-1}), which declined to 0.50 mg g^{-1} VS h^{-1} for municipal refuse compost by Day 31. Iannotti et al. (1994) reported that oxygen respirometry was highly correlated with composting time ($r = 0.80$, $P = 0.01$).

The Dewar flask self-heating test (Niese, 1963; Brinton et al., 1995) evaluates compost stability based on the rise over ambient temperature of compost in an insulated thermoflask. Richard and Zimmerman (1995) attempted to relate oxygen uptake to the maximum reheating potential but found that, whereas respiration rate clearly declined with the increasing age of the municipal refuse compost, temperature was less conclusive in predicting the stabilization process.

Cold Storage Effects

Although very little research has studied the effects of cold storage on compost samples (Gagnon et al., 1993), several researchers have investigated the effects of cold storage on soil samples. Munro and MacKay (1964) found that air-dried soil samples stored at 5°C gave nearly constant nitrate production during a 15-wk storage period. Samples stored at the same temperature but at 10 and 20% moisture showed decreasing nitrate production with increasing length of storage. Storrier (1966) concluded that low temperature storage of moist samples may minimize changes in nitrogen mineralization and biological activity. Bartlett and James (1980) concluded that microbial changes were minimized in refrigerated moist (32.8%) soil samples as compared with those which were air-dried and stored for up to 16 wk at either room temperature or frozen. Similarly, Ross et al. (1980) found that storage at 4°C for up to 56 d was more suitable for retaining biomass values for undried soil samples than storage at either 25°C or −20°C.

However, Zelles et al. (1991) found that air-drying soil samples prior to storage was more detrimental to retaining biological activity than storing moist samples at 4°C, −18°C, or −140°C up to 20 mo. Stenberg et al. (1998) found that microbial biomass and activity in moist soil samples suffered more pronounced effects under refrigeration for 13 mo at 4°C than at −20°C.

Degradation occurs because psychrotrophs tend to predominate in refrigerated foodstuffs (Prieto et al., 1991; Ken-Yuon and Torres, 1993; Cox et al., 1998), and may be at least partially responsible for the continuing humification of compost under refrigeration. Psychrotrophs are versatile and are able to grow over a wider temperature range than psychrophilic organisms (Russell, 1992). Herbert and Bhakoo (1979), citing Morita (1975), indicate that while psychrotrophs grow at 0°C, their maximum growth temperature exceeds 25°C.

Experimental Objectives

In this study we investigated the changes occurring in the following three variables with composting time and length of storage period: (i) Dewar flask self-heating capacity, (ii) oxygen uptake rate, and (iii) CEC. Based on the results, mathematical models were developed that predict change in the variables with time and storage at 4°C. In order to develop the models, these trends were followed throughout the entire composting process. Cold storage was included as a variable because very little investigation of its effects on compost samples has been conducted. At the present time, short-term cold storage at 4°C is recommended by the U.S. Composting Council when samples cannot be tested immediately (U.S. Composting Council, 1995).

MATERIALS AND METHODS

This study followed the composting process of a locally successful commercial composting operation that had been producing alkaline-stabilized biosolids compost in the Washington, DC metro region for 12 yr. The researchers did not control the composting operation or attempt to influence the course of the composting process. The objective of the research was to apply maturity indices to a commercial composting operation in order to assess the effectiveness of the indicators in a "real-world" setting.

Raw Materials

Composting was conducted using the Beltsville extended, aerated, static pile method. The piles were indoors and aerated using negative pressure. Vacuum-filtered, lime-stabilized biosolids (sewage sludge) from the Blue Plains sewage treatment plant in Washington, DC was mixed with wood chips in a ratio of 4:3 parts wood chips to biosolids by volume at the Washington Suburban Sanitary Commission's (WSSC) composting plant (Site 2) in Silver Spring, MD. The mixture was placed on a bed of woodchips containing aeration pipe and composted using the Beltsville static-aerated pile method (Willson et al., 1980). Aeration was monitored by oxygen meters and temperature was monitored by thermocouples placed at a 1.5-m depth from the surface at 6.1-m intervals in each pile.

Composting Method

Individual piles were 3.0 × 6.1 × 30 m (height × width × length) in dimension and contained approximately 549 m^3 of material. They were constructed side by side in rows that contained 20 piles per row (approximately three piles were constructed daily). The pile used for sampling was marked off by stakes to separate it from other piles. Two and three tenths meters of fresh material was bedded on 0.3 m of wood chips and covered by 0.5 m of aged compost as cover. The piles were indoors and aerated using negative pressure.

Composting was conducted in two stages. Days 1 through 28 comprised the "actively composting" pile (Stage I). After Day 28, the pile was torn down and sieved through a 12.7-mm sieve to remove large wood chips. The sieved material was moved to the curing building, where a new aerated pile (Stage II) was constructed.

In Stage I, samples were collected at a depth of 0.9 m from the surface. Samples (38 L each) were collected at five sampling locations, every 6.1 m along the 30-m length of the pile at locations where thermocouples had been placed.

The Stage II pile was constructed consisting of material from three actively composting piles, including the sampled pile. The sampled pile was staked off to differentiate it from the other two piles. The dimensions of the Stage II pile were

Table 1. Sampling and testing schedule for biosolids compost samples.

Sampling date	Week(s) of storage
5 Dec. 1997 (Day 1 of composting)	0, 3, 5, 8, 11
19 Dec. 1997 (Day 15)	1, 4, 7, 10
6 Jan. 1998 (Day 15)†	1, 4, 7, 10
20 Jan. 1998 (Day 29)	0, 4, 8, 11
27 Jan. 1998 (Day 36)	0, 3, 7, 11
3 Feb. 1998 (Day 43)	2, 5, 7, 10
10 Feb. 1998 (Day 50)	1, 3, 6, 8
17 Feb. 1998 (Day 57)	0, 2, 4, 6, 8

† Day 15 was duplicated over two piles.

3.0 × 3.0 × 6.1 m. Due to the much smaller dimensions of the Stage II pile, only three buckets of samples (57 L) were collected at a depth of 0.5 m in 1.2-m intervals per sampling date. After Day 30, the Stage II pile was torn down.

Sampling Methodology

Samples were collected biweekly from the Stage I pile (Days 1 and 15). Five buckets of samples (95 L) were collected per sampling date from the Stage II pile. Due to problems with odor, Stage I was torn down at Day 21 and used as additional cover for other piles. A second Stage I pile at the compost site was then sampled at its Day 15, and was followed through Stage II at Days 29, 36, 43, 50, and 57 (Table 1).

Samples collected from five locations within the pile were mixed together in a large cement mixer, and transferred to 6.5-L plastic bags for storage at 4°C until analysis. Subsamples of this composite material were tested periodically over 11 wk of storage. Composite samples facilitated timely analyses. Individual samples collected at each sampling date were mixed to create a composite sample, sieved through a 4.0-mm sieve, and air-dried. Duplicate subsamples were analyzed for each sampling date.

Test Methods

The biological volatile solids (BVS) content of the composts was obtained after combustion of oven-dry solids at 550°C for 12 h. The Dewar flask reheat test (Niese, 1963; Woods End Research Laboratory, 1993; Brinton et al., 1995) was performed in 2-L insulated Dewar flasks using min-max thermometers. Samples were sieved through a 4-mm sieve, moistened to approximately 50% moisture (wet weight), and placed in the Dewar flask. The maximum daily temperature was recorded as well as the daily ambient temperature over a 5-d incubation period.

The oxygen uptake rate was measured using a modified method based on Iannotti et al. (1993) and the U.S. Composting Council (1995). A refrigerator incubator was used instead of a water bath to incubate the samples at 37°C. Samples were sieved through a 4.0-mm sieve and brought to approximately 50% moisture prior to analysis.

Due to the high lime content of the biosolids compost, a double extraction procedure for calcareous soils using a 0.4 *M* sodium acetate–0.1 *M* sodium chloride solution followed by 1.0 *M* magnesium nitrate was followed to analyze the cation exchange capacity (Polemio and Rhoades, 1977).

Statistical Analysis

The experiment was designed as an incomplete factorial in which the relationship between the dependent variables (maximum rise over ambient temperature, $\log_{10}$ of the oxygen uptake rate, and CEC) and the fixed independent variables (time [expressed in days] and storage [expressed in weeks]) was examined. Because samples were collected from the same individual pile throughout the composting period, correlations among residuals were expected. Potential covariance among the residuals was checked using the repeated measures option in the Mixed procedure of SAS Version 6.12 (Littell et al., 1996). No significant correlation among residuals over time were found. Statistical analysis was performed using two-way mixed models (Littell et al., 1996).

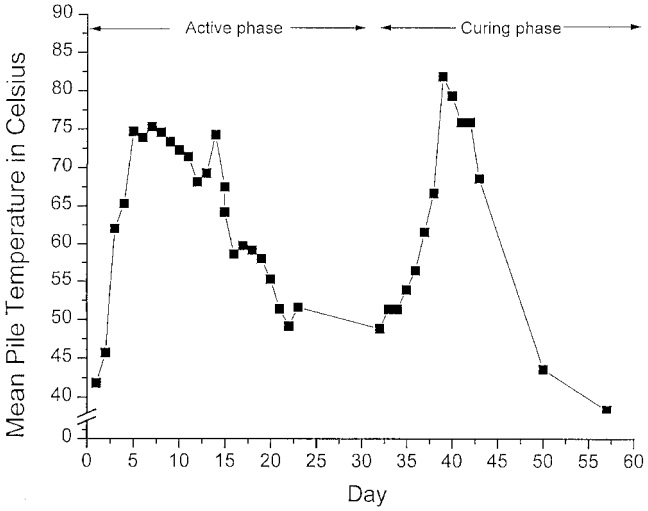

Fig. 1. Mean daily pile temperatures for biosolids compost for Stages I and II of composting.

RESULTS

Mean daily temperatures for Stages I and II of the compost pile are presented in Fig. 1. Stage I reached a mean maximum of about 75°C and droped off to about 50°C just before tear down. Stage II peaked at an even higher mean maximum temperature of about 85°C, and dropped off sharply thereafter to a mean temperature of about 40°C at the time the pile was torn down. The biological volatile solids (BVS) declined from 53.5 to 47.8% in 57 d (Fig. 2). Most of the decrease occurred during Stage I.

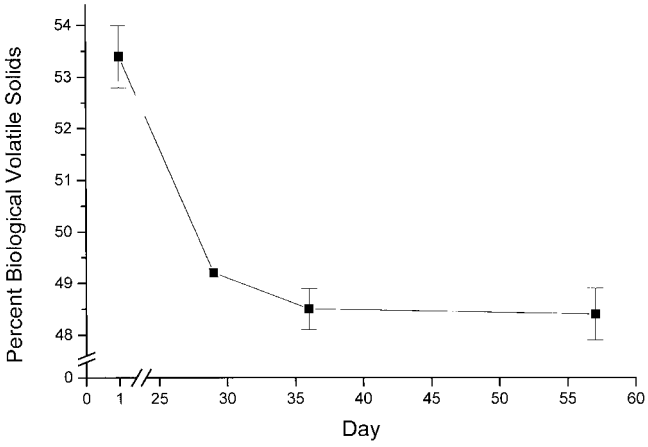

Fig. 2. Change in mean percent biological volatile solids (BVS) content of biosolids compost samples between Days 1 and 57 of composting.

Table 2. Solution for the effects of age (day) and storage for the maximum rise above ambient temperature in the Dewar flask for biosolids compost.

Effect	Estimate	Standard error	DF	*t*	$P < 0.05$
Intercept	50.5	1.32	58	38.3	0.0001
Day	−1.42	0.231	58	−6.13	0.0001
Day^2	0.0542	0.00917	58	5.91	0.0001
Day^3	-6.69×10^{-4}	1.00×10^{-4}	58	−6.67	0.0001
Storage	−0.0410	0.310	58	−0.13	0.8954
Day × storage	−0.0993	0.0158	58	−6.42	0.0001
Day^2 × storage	0.00156	3.41×10^{-4}	58	4.58	0.0001

Dewar Flask Self-Heating Test

Analysis of the mean maximum temperature rise over ambient in the Dewar flask test revealed that age (expressed in days) had significant linear, quadratic, and cubic effects on the reheating capacity of the compost. Significant interaction effects between age and storage time also were found, and the final-third order polynomial model obtained through backward step-wise regression also contained quadratic and cubic effects due to age (Table 2).

A response surface generated from the model in Table 2 shows values for maximum temperature rise above ambient in the Dewar flask test for various age and storage values (Fig. 3). Storage had little effect on the maximum temperature rise over ambient for samples taken at Days 1 and 57. A rise of between 44.0 and 52.0°C over ambient is predicted for composts less than 7 d old and stored for up to 11 wk. For compost sampled after 40 d of composting, the predicted temperature rise is 35.0 to 40.0°C over ambient for up to 2 wk of storage, which would characterize it as very immature compost according to the classification system of Niese (1963) and Brinton et al. (1995). The 57-d-old compost would have a predicted rise of between 15.0 and 20.0°C.

Current standards consider a temperature rise of 10°C or less to be indicative of a stable compost. A temperature rise of between 10 and 20°C is indicative of moderately stable compost, while a temperature rise of above 20°C is indicative of actively composting materials (Brinton et al., 1995). Data indicate that between Days 1 and 57, samples may be stored for up to 2 wk with little decline in temperature. Samples collected at Day 1 may be stored for up to 12 wk with no significant decline in temperature, while those collected at Day 57 of composting can be stored for up to 8 wk before declining to the lower predicted temperature range.

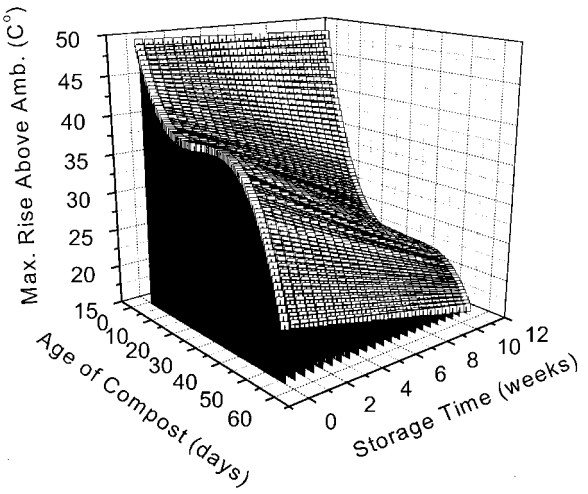

Fig. 3. Response surface depicting the predicted maximum rise above ambient temperature reached in the Dewar flask self-heating test for the age of the biosolids compost (expressed in days) and storage (weeks) at 4°C.

Oxygen Respirometry

Oxygen uptake rate is expressed as the $\log_{10}$ in mg O_2 g^{-1} BVS h^{-1}. Significant effects of oxygen uptake were found for both age and storage, with significant linear, quadratic, and cubic effects for age, and linear and quadratic effects for storage (Table 3).

The response surface generated from the model indicates that samples collected at Days 1 and 15 of composting are not affected by storage of up to 11 wk (Fig. 4). Samples collected during this interval would show no decline in oxygen uptake rate due to refrigerated storage according to the model. The storage effect appears to be most pronounced for samples collected after Day 29 and stored for several weeks. Data indicate that younger compost samples exhibit an oxygen uptake rate that is relatively robust with respect to cold storage effects. After Day 29, storage reduces the oxygen uptake, indicating that the compost is more mature than age alone would suggest.

Cation Exchange Capacity

Cation exchange capacity is expressed in cmol kg^{-1} of air-dried compost. The CEC increases with age and storage, an effect that is seen in all samples (Fig. 5). Significant linear and quadratic effects were found for both age and storage. A significant linear interaction was found between age and storage, as well as a significant linear by quadratic interaction between age and storage (Table 4).

The response surface (Fig. 5) based on the model shows that CEC is at its lowest predicted range (5.0 and 12.0 cmol kg^{-1}) at Days 1 through 5. For Storage Weeks 8 through 11 the CEC increases to 10.0 to 15.0, and by Storage Week 12, from 15.0 to 20.0 cmol kg^{-1}. At Day 57 of composting, the predicted CEC has increased to between 20.0 and 24.0 cmol kg^{-1} for samples tested with up to 1 wk of storage. At Day 57 the predicted CEC is from 20 to 24 cmol kg^{-1} for samples stored up to 1 wk. As storage time increases, CEC increases and then declines back to 20 to 24 cmol kg^{-1} at 11 wk of storage. This is reflective of the quadratic trend in the model, which makes interpretation difficult. Although an increase in CEC due to refrigerated storage can be attrib-

Table 3. Solution for the effects of age (day) and storage for the $\log_{10}$ of the oxygen uptake rate in mg O_2 g^{-1} biological volatile solids (BVS) h^{-1} for biosolids compost.

Effect	Estimate	Standard error	DF	*t*	$P < 0.05$
Intercept	0.467	0.0379	56	8.19	0.0001
Day	−0.0375	0.00396	56	−9.47	0.0001
Day^2	8.53×10^{-4}	1.63×10^{-4}	56	5.23	0.0001
Day^3	-7.13×10^{-6}	1.88×10^{-7}	56	−3.90	0.0003
Storage	0.0277	0.00878	56	3.15	0.0026
Storage^2	−0.00212	0.000796	56	−2.66	0.0102
Day × storage	0.000347	0.000464	56	0.75	0.4583
Day × storage^2	5.49×10^{-5}	1.35×10^{-5}	56	−4.06	0.0001

uted to continuing humification under refrigeration, the decline in CEC after 10 wk of storage seems to contradict this.

DISCUSSION

This study applied the maturity indices to a locally successful commercial composting operation that had been producing alkaline-stabilized biosolids compost in the Washington D.C. metro region for 12 yr prior to the start of this study. The purpose of this study was to follow the composting processes of commercial composting operations and test the maturity indices under these conditions. The researchers did not control the composting operation of the WSSC or attempt to influence the course of the composting process. Studies of this kind are common in ecological research, but less common in the agronomic sciences. This type of study was conducted because while a great deal of past composting research has focused on small-scale, researcher-controlled composting, the ultimate objective of the research is to apply maturity indices to commercial, state-run, or on-farm composting operations.

Due to time limitations, out of the many maturity tests that could have been selected for this study, we chose the Dewar flask self-heating capacity, oxygen respirometry, and CEC because these tests are comparatively simple and inexpensive for a compost site or laboratory to conduct, and are well attested to in the literature. While useful for determining the presence of volatile organic acids that might adversely affect plant growth, plant germination and/or growth response was not tested in this study. As Fig. 1 indicates, temperatures were above 65°C between Days 5 and 15 during Stage I of composting, and declined to about 50°C by the end of Stage I. Although there is some possibility that these high temperatures could have had a suppressive effect on the microflora and created inefficiencies in the process, this does not invalidate the sensitivity of the indicators that measured change throughout the process, regardless of whether maturity was achieved in absolute terms. High temperatures are frequently reached in large-scale composting systems (Willson et al., 1980; Sikora et al., 1981). This is often due to the geometry of pile construction. It is frequently the case that the larger the pile, the more difficult it is to achieve enough aeration to dissipate heat rapidly. Thus, heat will tend to build up and temperatures greater than 65°C are common. These are issues of scale that are rarely addressed in composting studies. Although it is possible that reinoculation did occur between Stages I and II, which might account for the high temperatures reached in Stage II, this would not affect the ability of the indicators to measure changes in the compost over time.

As determined by time parameters set by the WSSC, the compost was in "curing" (Stage II) after Day 25 (Fig. 1). Since we did not attempt to influence the composting

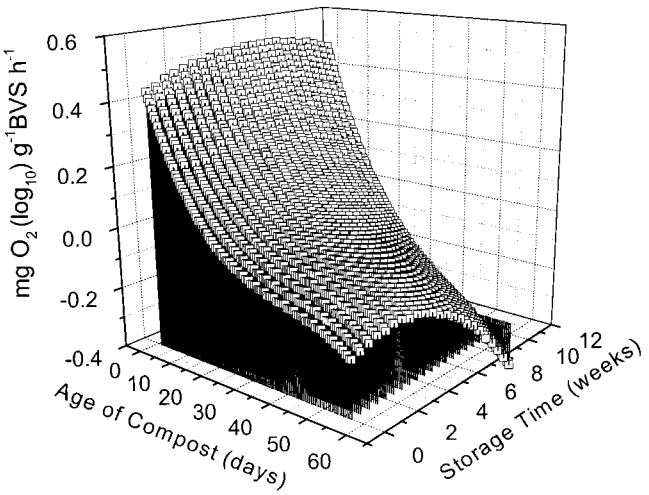

Fig. 4. Response surface depicting the predicted values of the $\log_{10}$ of the oxygen uptake rate for the age of the biosolids compost (expressed in days) and storage (weeks) at 4°C.

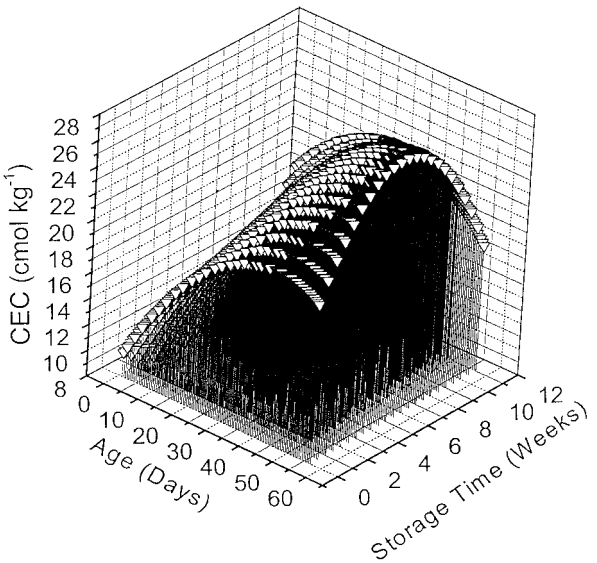

Fig. 5. Response surface depicting the predicted cation exchange capacity (CEC) values in cmol kg^{-1} for the age of the biosolids compost (expressed in days) and storage (weeks) at 4°C.

Table 4. Solution for the effects of age (day) and storage on cation exchange capacity (CEC) of biosolids compost.

Effect	Estimate	Standard error	DF	*t*	$P < 0.05$
Intercept	9.34	3.46	60	2.70	0.0091
Day	0.542	0.166	60	3.28	0.0017
Day^2	−0.00615	0.00248	60	−2.48	0.0161
Storage	−1.17	1.33	60	−0.87	0.385
Storage^2	0.168	0.118	60	1.42	0.162
Day × storage	0.0726	0.0384	60	1.89	0.0638
Day × storage^2	−0.00805	0.00365	60	−2.21	0.0313

operation of the WSSC, we used this terminology to describe Stage II, even if it was concluded that the end product was not fully mature by Day 57, or if the composting process itself was less than optimal. The results of the Dewar flask test revealed that by Day 57 the compost was not yet stable, and the CEC showed continuing humification during refrigerated storage over 12 wk. These two tests are effective maturity indicators even under suboptimal composting conditions. As shown in Fig. 2, BVS declined by 9.4% between Days 1 and 57, which is consistent with other researchers' findings for the composting of lime-stabilized biosolids (Sikora et al., 1981).

Richard and Zimmerman (1995) attempted to correlate the maximum reheating temperature with oxygen respirometry and found that the oxygen respiration rate showed a curvilinear decline from about 15 mg O_2 g^{-1} VS h^{-1} to 8 mg O_2 g^{-1} VS h^{-1} over three phases of controlled composting. The maximum reheating temperatures declined from 45 to 37°C, then increased to 42°C in the third phase.

In our study, however, the maximum rise above ambient temperature clearly declined with age over the 8-wk composting period to about 20°C for samples tested fresh. According to the stability criteria of Niese (1963) and Brinton et al. (1995), such compost would be classified as moderately stable. The model suggests that an additional week of curing may stabilize the compost to meet the 10°C "stable" criterion. The effect of cold storage at 4°C on samples varied with compost age. Changes in compost microbiology at different stages in the composting process might explain these interactions. Following the response surface, samples collected between Days 1 and 10 of composting are less affected by cold storage of up to 11 wk (Fig. 3). This indicates that the youngest samples are able to rebound from cold storage with little inhibition of microbial activity. Day 15 seems to show a clear transition, in which samples collected after this time were affected by refrigerated storage. The refrigerated storage effect after Day 15 is clearly curvilinear, indicating that older samples are more strongly affected by cold storage, and lower maximum temperatures can be expected after 2 wk of refrigeration. Day 57 samples were not strongly affected by refrigerated storage, suggesting that readily available nutrients are depleted and mature or near-mature composts are not affected by refrigerated storage.

As temperatures decline and the readily available substrate is used up, actinomycetes and fungi become dominant. Actinomycetes and fungi are important in the degradation of cellulose and lignin, which are more resistant to attack (Polprasert, 1989, p. 63–80). There was less biological activity found in stored samples than in samples tested fresh, which may be due to the decrease in readily available substrate after Day 15 of composting (Fig. 3). Other reasons for reduced biological activity could be (i) the microorganisms were more vulnerable to storage effects and were less able to rebound after cold storage or (ii) a change in microbial population occurred and these organisms are less tolerant of cold storage. Storage effects on unstable composts are small but as composts mature, extended storage will alter the maximum temperature rise, suggesting a more stable compost than recorded for a compost tested fresh. It appears from the CEC data that biodegradation continues under refrigeration, possibly due to the activity of psychrotrophs, which may metabolize the substrate under refrigeration (Prieto et al., 1991; Ken-Yuon and Torres, 1993; Cox et al., 1998).

After 29 d of composting the $\log_{10}$ of the oxygen uptake rate was essentially constant (Fig. 4). These data indicate that this test is not sensitive to change throughout the entire composting process and is not adequate as a sole stability indicator. If this test were used exclusively, it would lead the producer to conclude that the compost had stabilized halfway through the process (Iannotti et al., 1993, 1994), a conclusion that, based on results of the Dewar flask and CEC tests, is clearly erroneous. Application of immature or unstable compost to soil systems can lead to the depletion of available nitrogen and/or the release of volatile organic acids, which are the by-products of the compost's continuing decomposition in the soil. Iannotti et al. (1993) found an initial high rate of oxygen consumption (2.0 mg g^{-1} volatile solids h^{-1}), which declined to 0.50 mg g^{-1} VS h^{-1} for municipal refuse compost by Day 31. Since the composting process was apparently only monitored for 31 d, it would appear that further curing, as indicated by the water extract organic C to organic N ratio of 7.7 (Iannotti et al., 1993), was needed. Similarly, in this study, our values for oxygen uptake declined from about 3.30 mg g^{-1} BVS h^{-1} to about 0.50 mg g^{-1} BVS h^{-1} about Day 25 of composting. Based on the ratio of organic C to organic N in the water extract, which was above the threshold value of 5.88 (Chanyasak et al., 1982), Iannotti concluded that further curing was needed. In our study, the Dewar flask test showed continuing heating above the threshold value of 10°C (Brinton et al., 1995), and we concluded that readily available carbon was still present and was responsible for the continued heating at the end of the composting process. Results of the CEC test also showed continuing change in

the values at the end of the composting process. Because both the CEC and Dewar flask tests showed continued change at the end of the composting process, we conclude that oxygen respirometry may not be an adequate test for Stage II of composting.

Differences between the Dewar flask and oxygen respirometry tests are probably not due to exothermic chemical reactions in the lime-stabilized sludge because although an exothermic reaction occurs quickly when highly reactive Ca materials are mixed with water, it is of relatively short duration (approximately 30 min; Sikora and Francis, 2000). It is likely that the exothermic reaction occurs when $Ca(OH)_2$ is added at the treatment plant during the lime stabilization process and the heat is dissipated before the biosolids arrives at the compost facility. The pH of the lime-stabilized biosolids may still be elevated at pH 11 at the start of composting but, according to laboratory simulation studies, pH drops below 8.0 because CO_2 production was detected after 4 d (Sikora et al., 1983). The calcination reaction neutralized $Ca(OH)_2$, producing $CaCO_3$, which would not be sufficiently reactive to result in an exothermic reaction. Therefore, an exothermic reaction may influence the self-heating test for the 1-d-old samples taken in this study, but samples taken later would not have sufficient reactive Ca to interfere with the stability test. Although mean pile temperatures rose to greater than 65°C between Days 5 and 15 in the composting pile and after Day 38 in the curing pile, these elevated pile temperatures did not appear to affect the results of the Dewar flask study. Day 57 compost samples tested without cold storage showed a 15°C rise above ambient temperature, which suggests that microflora were not destroyed as a result of high temperature.

Cation exchange capacity clearly increases with the age of the compost and storage time (Fig. 5). These results appear to indicate that humification continues during storage. During the humification process, complexation and condensation reactions occur, producing high molecular weight, fairly stable compounds (Lax et al., 1986). Either the storage temperature is not sufficiently low to completely inhibit the activity of composting microorganisms, or psychotrophic microorganisms, which are able to metabolize the substrate at lower temperatures, become more competitive under refrigeration. If an ecological transition is occurring between these two communities, then the shift from one active population to another would provide the basis for continuing humification.

Overall, CEC was found to increase from about 6 to about 24 cmol kg^{-1}, at its maximum, an increase of fourfold (Fig. 5). Harada and Inoko (1980) considered a municipal refuse compost with a CEC of greater or equal than 60 cmol kg^{-1} expressed on an ash-free basis to be sufficiently mature for application to the soil. However, these researchers found a smaller increase for municipal refuse compost (40 to 60 cmol kg^{-1}), an increase of 1.5 times the starting value. Unlike the present study, which used unground samples, they used a finely ground sample (60 mesh or smaller), which had a starting value much higher than that of an unground compost. The CEC values are affected by both preparation techniques and the nature of the substrate, suggesting that one threshold CEC value for maturity is not possible.

There does not appear to be a consensus about the best method for long-term storage, but storage of moist samples at low temperature minimizes disruption of microbial biomass (Bartlett and James, 1980; Ross et al., 1980). One can conclude that during the long-term refrigerated storage of moist compost samples slow microbial degradation of the substrate occurs. Since psychrotrophs are able to metabolize substrate more efficiently at lower temperatures than mesophiles (Margesin and Schinner, 1994; Cox et al., 1998), they could eventually predominate under long-term cold storage conditions. Although degradation may be slower at 4°C than at ambient, under long-term storage degradation can be significant and alter the substrate, changing the test results.

CONCLUSIONS

The Dewar flask self-heating test was the most useful indicator of compost maturity. This test showed change throughout the 57-d composting period while oxygen respirometry was not sensitive to change after 29 d of composting, suggesting that this test is not by itself an adequate indicator of stability. Although CEC was found to increase substantially for stored samples, no clear trend was seen for samples that were tested without storage. Storage primarily affected samples that were more than 15 d old. Storage effects varied for different tests. Results of this study have yielded potentially useful information about age and storage time interactions on the three variables tested. Since it is not always possible to test compost samples fresh from the pile, response surfaces can show how long samples may be stored before being affected by refrigerated storage.

Cation exchange capacity values recorded for biosolids composts suggest that each compost substrate should be treated as unique, and maturity indicator data collected be cross-referenced with values reported in the literature for the same compost produced under similar conditions.

Results of this study indicate the necessity of timely analysis in order to avoid the problem of prolonged sample storage. Since this problem appears to be underreported in the literature (Wu and Ma, 2001), our results provide information on changes in indicator values in samples refrigerated for up to 3 mo. Because these results were obtained from a single biosolids compost operation, future studies should test additional biosolids compost to determine whether the age and storage relationship modeled in this study can be applied to biosolids compost or all composts.

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